

PHOTOSENSITIVE RESIN COMPOSITION

CROSS REFERENCE TO RELATED APPLICATION

This application is based on Korean patent application No. 10-1999-
5 0033884 filed on August 17, 1999, which is incorporated hereinto by reference.

BACKGROUND OF THE INVENTION**(a) Field of the Invention**

The present invention relates to a light-sensitive resin composition capable of being developed by an alkaline aqueous solution, more particularly
10 to a photosensitive resin composition applicable to a color filter and for thin film transistor circuit passivation in the liquid crystal display manufacturing process.

(b) Description of the Related Art

Demand for photosensitive resins capable of being developed by an alkaline aqueous solution is recently increasing in many fields requiring micro
15 image formation due to the so-called photolithography technology development in which images are formed using active lights such as ultraviolet rays, etc.

Alkaline developing type photosensitive resins have been used in dry film resists for printed circuit board fabrication, photoresists for semiconductor circuit fabrication, etc., and their applications such as color filter and various
20 circuit passivation are recently being expanded into semiconductors and flat panel display fields, including liquid crystal displays.

A photosensitive resin composition capable of being developed by an alkaline aqueous solution generally comprises a) a binder resin which is dissolved or swelled by an alkaline solution; b) a crosslinking compound having
25 at least two ethylenically unsaturated bonds; c) a photopolymerization initiator; and d) a solvent capable of dissolving the above mentioned constituents, in which dyestuff, pigment, or various additives which improve the filming property or adhesive property to the substrate can be contained if necessary.

The above a), as a binder resin which is dissolved or swelled by an

alkaline aqueous solution, generally comprising a structure containing carboxylic acid or a carboxylic acid anhydride or hydroxide group, an amino group, an amide group in the polymer chains, and a novolak based phenol resin, an acryl based resin homopolymer, or an acryl based resin copolymer, is widely
5 used.

Particularly, an acryl based binder resin having superior transparency for visible light rays is widely being used when the photosensitive resin composition is used as a color filter and for thin film transistor circuit passivation in the flat panel display fields.

10 Copolymers using one or more compounds selected from the group consisting of acrylic acid, methacrylic acid, an alkyl ester thereof, and substituted or unsubstituted aryl esters are mainly used as acryl based binder resins soluble in the alkaline solution.

The use of benzyl acrylate/methacrylic acid copolymer is disclosed in
15 U.S. Patent Nos. 4,629,680 and 4,139,391, methyl methacrylate/2-ethylhexyl methacrylate/methacrylic acid terpolymer is disclosed in Japanese Patent Publication No. Showa 54-34327, and methyl methacrylate/ethyl acrylate/acrylic acid terpolymer is disclosed in Japanese Patent Publication No. Showa 55-6210. Furthermore, a copolymer using allylacrylate, hydroxyalkyl acrylate, methacrylic
20 acid, etc. is disclosed in Japanese Patent Laid-open Publication No. Heisei 9-23059.

A compound of the above b), as a crosslinking compound having at least two ethylenically unsaturated bonds, includes polyethylene glycol di(meth)acrylate, propylene glycol di(meth)acrylate, 1,3-butanediol
25 di(meth)acrylate, neopentyl glycol di(meth)acrylate, 1,6-hexanediol di(meth)acrylate, trimethylolpropane tri(meth)acrylate, pentaerythritol tetra(meth)acrylate, dipentaerythritol penta(meth)acrylate, pentaerythritol hexa(meth)acrylate, etc.

In photosensitive resin compositions of existing technologies, this
30 crosslinking compound is simply distributed in a binder resin of the composition, and when it is exposed to an active light such as ultraviolet rays, etc., a crosslinking reaction occurs and a network structure is formed preventing an

alkaline soluble resin from dissolving by a developer in the development process, consequently playing a role in the leaving of images on a substrate.

However, solubility differences between areas exposed to light and areas not exposed to light in existing photosensitive resin compositions are not
5 large enough, and binder resin which should practically remain during the development process is partially dissolved by a development solution, and consequently it is difficult to obtain desirable micro patterned shapes in most cases.

On the other hand, when crosslinking compounds are excessively used
10 to prevent this phenomena, not only are process properties decreased by surface hardness deterioration after exposure to light, but also process yields decrease as an exposing amount to light causing sufficient crosslinking reaction increases.

Furthermore, there is a disadvantage in this case that solubilities of the
15 non-exposed areas are also deteriorated, thus reducing the resolving power as a resist.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide a photosensitive resin which has superior resolving power by having a chemical bond between a
20 binder resin and a crosslinking compound as well as a chemical bond between binder resin chains at an exposed area during an exposing process, thus maximizing the solubility difference between an exposed area and a non-exposed area during the developing process, and which has process benefits as well as superior film characteristics by reducing the consumed amount of
25 crosslinking compound, thus decreasing total exposed light quantity in order to solve the above described problems.

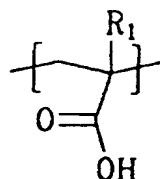
The present invention provides a photosensitive resin composition comprising a self-curable binder resin, a photopolymerization initiator, a crosslinking compound having at least two ethylenically unsaturated bonds, and
30 a solvent, wherein the self-curable binder resin is the following Chemical Formula 1:

[Chemical Formula 1]

- A - B - C -

where A is a compound represented as in the following Chemical Formula 1-A;

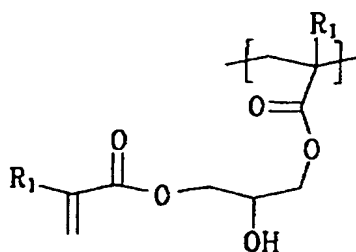
[Chemical Formula 1-A]



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B is a compound represented as in the following Chemical Formula 1-B;

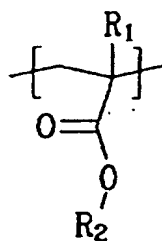
[Chemical Formula 1-B]



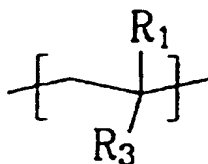
C is a compound represented as in the following Chemical Formula 1-C

10 and / or Chemical Formula 1-C';

[Chemical Formula 1-C]



[Chemical Formula 1-C']



15 wherein R_1 is H or CH_3 , R_2 is an alkyl group having 1 to 8 carbon atoms, a hydroxy group substituted alkyl group, or a substituted or unsubstituted aryl group having 1 to 12 carbon atoms, and R_3 is a benzene, an alkyl group having 1 to 8 carbon atoms, an alkoxy group having 1 to 8 carbon atoms, a benzene

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having a $C_1 \sim C_6$ alkyl substituent, a benzene having a $C_1 \sim C_8$ alkoxy substituent, or a hydroxide group or a halogen substituted benzene.

The A portion in the binder resin of Chemical Formula 1 accounts for 10 to 50 mol%, the B portion accounts for 0 to 15 mol%, and the C portion
5 accounts for 50 to 90 mol% of the total binder resin.

A crosslinking compound having at least two unsaturated groups can further be used in the photosensitive resin composition if necessary.

A self-curable binder resin of the Chemical Formula 1 is obtained by reacting a copolymer comprising a constituent of a compound having one or
10 more carboxylic acids selected from the group consisting of the following Chemical Formulae 2, 3, and 4 with a compound of the following Chemical Formula 5.

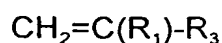
[Chemical Formula 2]



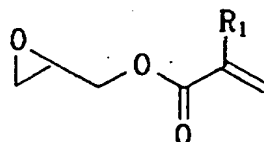
15 [Chemical Formula 3]



[Chemical Formula 4]



[Chemical Formula 5]



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wherein R_1 is hydrogen or a methyl group, R_2 is a compound selected from the group consisting of an alkyl group having 1 to 8 carbon atoms, a hydroxy group substituted alkyl group, and a substituted or unsubstituted aryl group or arylalkyl group having 4 to 12 carbon atoms, and R_3 is a compound
25 selected from the group consisting of a benzene, an alkyl group having 1 to 8 carbon atoms, an alkoxy group having 1 to 8 carbon atoms, a benzene having a $C_1 \sim C_6$ alkyl substituent, a benzene having a $C_1 \sim C_8$ alkoxy substituent, and a hydroxide group or halogen substituted benzene.

A copolymer which is prepared by copolymerizing a self-curable binder

resin of the Chemical Formula 1 with one or both of a compound of the Chemical Formula 2 and a compound of the Chemical Formula 3 or Chemical Formula 4 in a certain mole ratio is put into a flask with a stirrer and a nitrogen injecting hole attached, and then dissolved using a solvent such as methyl ethyl ketone, etc. A number average molecular weight of a copolymer to be used is 1,000 to 100,000 and preferably 2,000 to 30,000.

After increasing the flask temperature to 120 °C and slowly adding a compound of Chemical Formula 5 over one hour, the solution is reacted until an epoxy group is completely removed. A self-curable binder resin is prepared by forming precipitates from the reactant using a mixture with a ratio of n-hexane and methanol of 1:1, and drying the precipitates under vacuum.

An epoxy group of a compound of Chemical Formula 5 is reacted with carboxylic acid existing in the binder resin and forming an ester consequently forming a self-curable binder resin, wherein a reactive (meth)acryl group is employed in the linear binder resin as a branch.

This obtained self-curable binder resin, as a resin which is dissolved or at least swelled by an alkaline aqueous solution, can cause the curing reaction by using an active light such as ultraviolet rays, etc.

Furthermore, the present invention provides a photosensitive resin composition prepared by mixing a copolymer in which a compound of the Chemical Formula 2 is copolymerized with one or both compounds of a compound of the Chemical Formula 3 and a compound of the Chemical Formula 4, a photopolymerization initiator, and a solvent.

While existing photosensitive resin components form a network structure by chemical reactions which occurs between only crosslinking compounds each other in UV exposure process, the photosensitive resin composition of the present invention forms a network structure not only with the crosslinking compounds but also with binder resin chains. Because of this, the photosensitive resin composition of the present invention provides the maximized solubility difference between an exposed area and a non exposed. Thus it can be obtained the superior film characteristics as well as the excellent

sensitivity and resolution by reducing the amount of crosslinking compound or not using it.

Acrylic acid, methacrylic acid, etc. can be used as a compound of the Chemical Formula 2, and its consumed amount is from 10 to 90 mol% based on the mole sum of total monomers in the copolymer.

A compound of the Chemical Formula 3 is preferably a compound selected from the group consisting of benzyl(meth)acrylate, phenyl(meth)acrylate, cyclohexyl(meth)acrylate, methyl(meth)acrylate, ethyl(meth)acrylate, or 2-ethylhexyl(meth)acrylate, and more preferably benzyl(meth)acrylate or phenyl(meth)acrylate.

A compound of the Chemical Formula 4 is preferably a compound selected from the group consisting of styrene, 4-hydroxystyrene, 4-methylstyrene, or vinyl acetate.

Furthermore, a compound of the Chemical Formula 5 is preferably glycidyl acrylate or glycidyl methacrylate. This compound uses 0.1 to 90 mol% of the carboxylic acid contained monomer in Chemical Formula 2.

A crosslinking compound having at least two unsaturated groups which can be used in the present invention if necessary includes a compound selected from the group consisting of polyethylene glycol di(meth)acrylate, propylene glycol di(meth)acrylate, 1,3-butanediol di(meth)acrylate, neopentyl glycol di(meth)acrylate, 1,6-hexanediol di(meth)acrylate, trimethylolpropane tri(meth)acrylate, pentaerythritol di(meth)acrylate, pentaerythritol tri(meth)acrylate, pentaerythritol tetra(meth)acrylate, pentaerythritol hexa(meth)acrylate, dipentaerythritol penta(meth)acrylate, dipentaerythritol hexa(meth)acrylate, trimethylolpropane triacrylate, and a mixture thereof, and preferably includes dipentaerythritol penta(meth)acrylate and dipentaerythritol hexa(meth)acrylate.

The content of the crosslinking compound is from 0 to 200 weight%, and preferably from 0 to 150 weight%, based on the total weight of the self-curable binder resin.

A photopolymerization initiator example is a compound selected from the group consisting of benzophenones such as benzophenone and

acetophenone; substituted benzophenones such as bis-4,4'-dimethylaminobenzophenone and bis-4,4'-diethylaminobenzophenone; triazine based photoinitiators such as 2,4,6-tris(trichloromethyl)-triazine; ketone based photopolymerization initiators such as N-methyl-2-benzoylmethylene- β -naphthothiazole and 2,2-dimethoxy-1,2-diphenylethanone; and a mixture thereof.

The content of the photopolymerization initiator is preferably from 0.1 to 10 weight% based on the total weight of the photopolymerizable composition.

A general solvent mainly used in the polymerization of acryl polymer, is a compound selected from the group consisting of methylethylketone, cyclohexanone, tetrahydrofuran, methyl cellosolve, methyl cellosolve acetate, dimethyl formamide, propylene glycol methylether acetate, 2-methoxyethylether, and a mixture thereof.

On the other hand, small amounts of a pigment, a dyestuff, an antifoaming agent for increasing filming properties, a surfactant, a thermal polymerization preventing agent, an adhesion promoter, etc. in addition to the above basic components can be used in a photosensitive resin composition of the present invention if necessary.

The finally obtained photosensitive resin composition solution is filtered using a membrane filter having 0.1 to 5 μm pores. This filtered resin composition is filmed using a notified method such as a spin coating method, a roll coating method, a spray coating method, etc. A glass plate or a silicone wafer can be used as a filming substrate, wherein the film surface thickness is determined by filming conditions such as composition viscosity, concentration of solid content, filming rate, etc., and a 0.1 to 500 μm thick thin film can be obtained using a composition of the present invention.

The obtained thin film is pretreated by maintaining a temperature of 50 to 150 $^{\circ}\text{C}$ at a heating plate or an oven for 10 to 500 seconds.

Ultraviolet rays are irradiated on the somewhat dried thin film through a positive type test photomask (made by Toppan Printing Co., Ltd.), wherein the ultraviolet ray light is irradiated in the intensity of illumination of from about 30 to 500 mJ/cm^2 using a 1 kW high pressure mercury lamp, etc. containing g, h, and

i lines, and particular optical filters are not used.

The solubility of the ultraviolet ray irradiated area becomes far less than that of the non-ultraviolet ray irradiated area, thus maximizing the solubility difference between them.

- 5 The ultraviolet ray irradiated thin film is developed by a spray method or a dipping method at a temperature of 20 to 30 °C, wherein a KOH aqueous solution having from 9 to 12 pH or 0.1 to 5 weight% of tetramethylammonium hydride aqueous solution can be used as a developer.

DETAILED DESCRIPTION OF THE INVENTION

- 10 In the following detailed description, only the preferred embodiments of the invention have been shown and described, simply by way of illustration of the best mode contemplated by the inventor(s) of carrying out the invention. As will be realized, the invention is capable of modification in various obvious respects, all without departing from the invention. Accordingly, the description
15 is to be regarded as illustrative in nature, and not restrictive.

- Preferable SYNTHESIZING EXAMPLES and EXAMPLES are described as follows in order to help understand the present invention. However, the following SYNTHESIZING EXAMPLES and EXAMPLES are only for help understand the present invention, and the present invention is not
20 limited to the following SYNTHESIZING EXAMPLES and EXAMPLES.

[SYNTHESIZING EXAMPLE 1]

- 10 g of copolymer in which the mole ratio of benzylmethacrylate : methacrylic acid is 60 : 40, and the number average molecular weight is 10,000 were put into a flask with a stirrer and a nitrogen injecting hole attached and
25 dissolved using 100 μ m of methyl ethyl ketone. After increasing the flask temperature to 120 °C and slowly adding 0.3 g of glycidyl methacrylate over one hour, the solution was reacted until epoxy groups were completely removed. Precipitates were formed from the reactant using a mixture of a ratio of n-hexane and methanol of 1:1, and dried under vacuum thereby obtaining a
30 binder resin.

[SYNTHESIZING EXAMPLE 2]

A binder resin was obtained according to the method of SYNTHESISING EXAMPLE 1 except that 10 g of copolymer in which the mole ratio of benzylmethacrylate : methacrylic acid was 50 : 50 and the number
5 average molecular weight was 15,000 were used, and 0.6 g of glycidyl acrylate were also used.

[SYNTHESIZING EXAMPLE 3]

A binder resin was obtained according to the method of SYNTHESISING EXAMPLE 1 except that 10 g of copolymer in which the mole
10 ratio of benzylmethacrylate : methacrylic acid was 70 : 30 and the number average molecular weight was 15,000 were used, and 0.05 g of glycidyl acrylate were also used.

[SYNTHESIZING EXAMPLE 4]

A binder resin was obtained according to the method of
15 SYNTHESISING EXAMPLE 1 except that 10 g of copolymer in which the mole ratio of benzylmethacrylate : methacrylic acid was 70 : 30 and the number average molecular weight was 10,000 were used, and 0.04 g of glycidyl acrylate were also used.

[SYNTHESIZING EXAMPLE 5]

20 A binder resin was obtained according to the method of SYNTHESISING EXAMPLE 1 except that 10 g of terpolymer in which the mole ratio of ethylmethacrylate : methacrylic acid : styrene was 50 : 40 : 10 and the number average molecular weight was 14,500 were used, and 0.3 g of glycidyl acrylate were also used.

25 [SYNTHESIZING EXAMPLE 6]

A binder resin was obtained according to the method of SYNTHESISING EXAMPLE 1 except that 10 g of terpolymer in which the mole ratio of ethylmethacrylate : methacrylic acid : styrene was 60 : 30 : 10 and the number average molecular weight was 14,500 were used, and 0.05 g of glycidyl
30 acrylate were also used.

A photosensitive resin composition of the present invention was prepared using a binder resin prepared in the above SYNTHSIZING

EXAMPLES 1 to 6.

[EXAMPLE 1]

A photosensitive resin composition was prepared in compositions represented as in the following Table 1.

5 [Table 1]

A binder resin prepared in SYNTHESIZING EXAMPLE 1	5 g
Bis-4,4'-diethylaminobenzophenone	2 g
Propylene glycol methyl ether acetate	20 g

The above prepared composition solution was filtered using a Teflon membrane filter having 0.2 μm pores. A photosensitive resin composition was coated on a glass plate using a spin method, put on a heating plate, and maintained at a temperature of 80 $^{\circ}\text{C}$ for three minutes. Subsequently, after putting a positive type test photomask (made by Toppan Printing Co., Ltd.) as a contacting method on a thin film, ultraviolet rays were irradiated thereon, wherein ultraviolet ray light was irradiated at the intensity of illumination of 100 mJ/cm² using a 1 kW high pressure mercury lamp containing g, h, and i lines, and particular optical filters were not used. The ultraviolet ray irradiated thin film was developed by dipping it into a KOH aqueous solution developer having a pH of 10.5, for two minutes. After washing the thin film coated glass plate with distilled water, it was dried by blowing nitrogen gas and heated in a heating oven at 250 $^{\circ}\text{C}$ for one hour. The obtained film thickness was 3.5 μm and clear patterns having 8 μm of line width and gap could be obtained.

[EXAMPLE 2]

A photosensitive resin composition was prepared in compositions represented as in the following Table 2.

[Table 2]

A binder resin prepared in SYNTHESIZING EXAMPLE 2	5 g
Bis-4,4'-diethylaminobenzophenone	2 g
Propylene glycol methyl ether acetate	20 g

A film was formed from the above prepared photosensitive resin

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composition in a method as in EXAMPLE 1, exposed at the intensity of illumination of 100 mJ/cm², and developed.

The obtained film thickness was 3.5 μm and clear patterns having 7 μm of line width and gap could be obtained.

5 [EXAMPLE 3]

A photosensitive resin composition was prepared in compositions represented as in the following Table 3.

[Table 3]

A binder resin prepared in SYNTHESIZING EXAMPLE 3	5 g
Bis-4,4'-diethylaminobenzophenone	2 g
Propylene glycol methyl ether acetate	20 g

10 A film was formed from the above prepared photosensitive resin composition in a method as in EXAMPLE 1, exposed at the intensity of illumination of 150 mJ/cm², and developed.

The obtained film thickness was 4.0 μm and clear patterns having 9 μm of line width and gap could be obtained.

15 [EXAMPLE 4]

A photosensitive resin composition was prepared in compositions represented as in the following Table 4.

[Table 4]

A binder resin prepared in SYNTHESIZING EXAMPLE 4	4 g
Bis-4,4'-diethylaminobenzophenone	2 g
Propylene glycol methyl ether acetate	30 g
Pentaerythritol tetramethacrylate	4 g

20 A film was formed from the above prepared photosensitive resin composition in a method as in EXAMPLE 1, exposed at the intensity of illumination of 150 mJ/cm², and developed.

The obtained film thickness was 4.5 μm and clear patterns having 8 μm of line width and gap could be obtained.

25 [EXAMPLE 5]

A photosensitive resin composition was prepared in compositions represented as in the following Table 5.

[Table 5]

A binder resin prepared in SYNTHESIZING EXAMPLE 5	5 g
Bis-4,4'-diethylaminobenzophenone	2 g
Propylene glycol methyl ether acetate	20 g

- 5 A film was formed from the above prepared photosensitive resin composition in a method as in EXAMPLE 1, exposed at the intensity of illumination of 150 mJ/cm², and developed.

The obtained film thickness was 3.8 μm and clear patterns having 9 μm of line width and gap could be obtained.

10 [EXAMPLE 6]

A photosensitive resin composition was prepared in compositions represented as in the following Table 6.

[Table 6]

A binder resin prepared in SYNTHESIZING EXAMPLE 6	5 g
Bis-4,4'-diethylaminobenzophenone	3 g
Propylene glycol methyl ether acetate	40 g
Pentaerythritol tetraacrylate	2 g

- 15 A film was formed from the above prepared photosensitive resin composition in a method as in EXAMPLE 1, exposed at the intensity of illumination of 100 mJ/cm², and developed.

The obtained film thickness was 3.1 μm and clear patterns having 7 μm of line width and gap could be obtained.

20 [EXAMPLE 7]

- A photosensitive resin composition was prepared in compositions represented as in the following Table 7. A photosensitive resin composition was prepared not by synthesizing a binder resin first, but by putting the starting material together with constituents comprising the photosensitive resin composition in the present EXAMPLE 7, different from the above EXAMPLES.
- 25

[Table 7]

Benzylmethacrylate/methacrylic acid copolymer (mole ratio = 60/40, number average molecular weight = 10000)	5 g
Glycidyl methacrylate	0.15 g
Bis-4,4'-diethylaminobenzophenone	2 g
Propylene glycol methyl ether acetate	20 g

A film was formed from the above prepared photosensitive resin composition in a method as in EXAMPLE 1, exposed at the intensity of 5 illumination of 150 mJ/cm², and developed.

The obtained film thickness was 3.6 μm and clear patterns having 8 μm of line width and gap could be obtained.

[COMPARATIVE EXAMPLES 1 to 3]

A photosensitive resin composition was prepared in compositions 10 represented as in the following Table 8.

[Table 8]

Benzylmethacrylate/methacrylic acid copolymer (mole ratio = 70/30, number average molecular weight = 10000)	5 g
Pentaerythritol tetra(meth)acrylate	5 g
Bis-4,4'-diethylaminobenzophenone	2 g
Propylene glycol methyl ether acetate	40 g

The results were obtained as in the following Table 9 by forming patterns as changing exposed light quantity after forming a film using the above 15 obtained photosensitive resin composition in a method as in EXAMPLE 1.

[Table 9]

Classification	COMPARATIVE EXAMPLE 1	COMPARATIVE EXAMPLE 2	COMPARATIVE EXAMPLE 3
Exposed light quantity	100 mJ/cm ²	150 mJ/cm ²	200 mJ/cm ²
Film thickness	-	3.0 μm	3.8 μm
Resolving power	Pattern lost	20 μm	10 μm

According to the results obtained from EXAMPLES 1 to 7, in the case of

forming a thin film using a photosensitive resin composition using a self-curable binder resin prepared in accordance with the present invention, the obtained thin film thickness was 3 to 5 μm and clear patterns having 7 to 9 μm of line width and gap could be obtained when the exposed light quantity was irradiated at the intensity of illumination of 100 to 150 mJ/cm^2 .

On the contrary, patterns themselves were lost when the exposed light quantity was 100 mJ/cm^2 in the case of photosensitive resin compositions of COMPARATIVE EXAMPLES 1 to 3 in which self-curable binder resins were not used, and the thin film thickness was 3.0 to 3.8 μm , and the line width and gap were decreased from 20 μm to 10 μm when the exposed light quantity was increased to 150 to 200 mJ/cm^2 . Therefore, it can be seen that clear patterns can be obtained as the exposed light quantity increases in the case of a photosensitive resin composition in which a self-curable binder resin is not used, and the increase of exposed light quantity means an increase in process time. Furthermore, it can be seen that clearer and finer patterns than the results of EXAMPLES according to the present invention could not be obtained since the thickness of the thin film pattern was represented as 3.8 μm and the line width and gap were represented as 10 μm even when the exposed light quantity was 200 mJ/cm^2 .

Therefore, the present invention can provide a photosensitive resin composition having process benefits, and superior resolving power and film properties since although less exposed light quantity is applied, a photosensitive resin composition using a self-curable binder resin can obtain much clearer and finer patterns compared with existing compositions, thus shortening time of UV exposure process.

While the present invention has been described in detail with reference to the preferred embodiments, those skilled in the art will appreciate that various modifications and substitutions can be made thereto without departing from the spirit and scope of the present invention as set forth in the appended claims.